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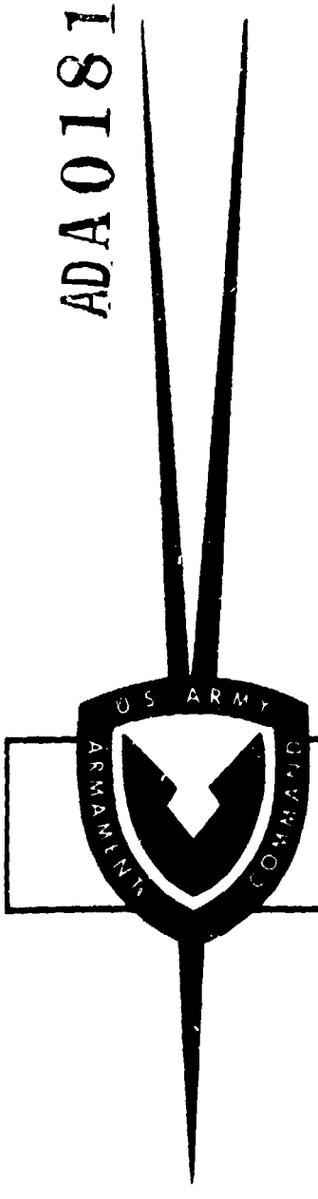
THE FORMATION OF REFRACTORY METAL ALLOYS BY SIMULTANEOUS DEPOSITION OF METALS FROM THE GASEOUS PHASE

ANDREW CROWSON

JULY 1974



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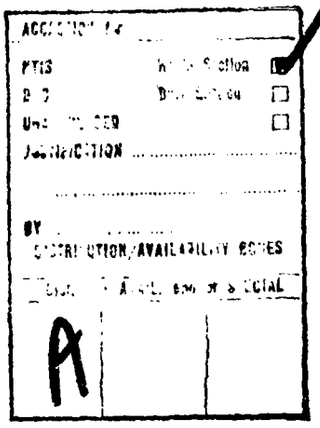

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Chemical vapor deposition investigations were undertaken to determine the feasibility of coating refractory metals and alloys onto the inside bores of gun barrels. Coatings of tungsten, molybdenum, vanadium, rhenium, and tantalum as well as tungsten alloys were attempted. Process parameters, compositions, and properties of the coatings were determined. Elemental tungsten depositions were initially used to establish the process parameters for subsequent work on tungsten alloy coatings. Ta-10W alloy was plated

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20. CONT

successfully on the steel substrate at 750°C and 1 atmosphere pressure. The coatings obtained were uniform and adherent. Properties of the alloy coating, Ta-10W, are such that this coating is especially applicable for a high temperature coating for gun barrels. (U) (CROWSON, ANDREW)

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INTRODUCTION

Erosion of gun barrels occurs when the gun barrel bore is exposed to the firing of a large number of rounds of ammunition. During firing, the gun barrel bore experiences conditions of high pressure, cycling temperature and short term corrosive environments. The combustion of the prevalent gun propellant, nitro cellulose, occurs according to the equation:[1]

$$2C_6H_5(OH)_2(NO_3)_3 \rightarrow 4CO_2 + 8CO + 6H_2O + H_2 + 3O_2 \qquad (1)$$

To withstand the environmental conditions set up during firing, an erosion resistant material should have the following characteristics: (1) high melting point, (2) high strength, (3) high specific heat, (4) thermal expansion comparable to that of the substrate material, (5) low compressibility, (6) resistance to embrittlement, and (7) wear resistance. In examining the physical and mechanical properties of available materials, the refractory metals -- molybdenum, niobium, tantalum, rhenium, vanadium and tungsten -- have many of these properties. The greatest problem in using these existing materials, however, is that of finding an economical process for successfully applying them as coatings.

Chemical vapor deposition is a method of plating in which the deposits are produced by heterogeneous gas-solid or gas-liquid chemical reactions at the surface of a substrate.[2] A volatile compound of the element or substance to be deposited is vaporized and then thermally decomposed, or reacted with other gases or vapors, at the substrate to yield nonvolatile reaction products which deposit on the substrate as a coating. It has the basic advantage of applying the refractory metals at temperatures well below the melting point of the coating element. A number of high melting point materials (tungsten, beryllium, chromium, cobalt, vanadium, boron, carbides, borides, and oxides) which are difficult to apply by other techniques have been successfully applied by chemical vapor deposition. Most refractory materials can be obtained in a pure state by vapor-deposition and considerable control can be exerted over the properties of the materials to be deposited.[3]

[1] Meyer, Karl H. and Gehring, John W., "A New Technique for Prevention of Erosion of Gun Barrels," AC Electronics-Defense Research Laboratories. Contract F08635-68-C-0044.

[2] Blocher, John M. and Withers, James C., Chemical Vapor Deposition, Second International Conference, New York, 1970.

[3] Powell, Carroll F., Oxley, Joseph H. and Blocher, John M., Vapor Deposition, John Wiley and Sons, Inc., New York, 1966.

Hydrogen reduction of a metal halide is one of the most frequently used types of chemical vapor deposition techniques. The reduction is carried out at elevated temperatures because a favorable thermodynamic reaction or a suitably rapid reaction rate is attained. This temperature may range from about 100°C in the reduction of the platinum group metal carbonyl halides up to >1000°C for reduction of refractory metal halides. Excessively high temperatures may result in ineffective reduction due to dissociation of the hydrogen halide or other primary products of reaction.[4]

In the particular hydrogen-reduction reactions of this study, the thermodynamics must be such that the following generalized reaction can proceed:

$$MX(g) + 1/2\ H_2(g) \rightarrow M(s) + HX(g) \qquad (2)$$

where M = metal to be deposited and X = halogen.

On the basis of thermodynamic principles, deposition temperatures can be calculated as a function of the ratio of gaseous reactants and products from equation (2). However, to obtain satisfactory metal deposits, careful consideration must also be given to time and temperature relationships, flow rates, composition of the reaction media, and other related practical factors. By the control of these parameters, an effective production procedure for depositing refractory metals and their alloys as coatings on gun tube bores can be attained.

The objective of this project was to chemical vapor deposit refractory metals and alloys as coatings on the bore of small arms gun barrels. Details of the process including substrate variables, process parameters and composition and properties of coatings were to be determined. The applicability of the deposits to improve the erosion and heat resistance of gun barrels and other weapon components was also to be studied.

EXPERIMENTAL PROCEDURE

Substrate

All coatings were deposited on Cr-Mo-V gun steel tubing. The nominal composition (in weight percent) of the gun steel is:

Carbon	0.41-0.49%	Silicon	0.20-0.35%
Manganese	0.60-0.90%	Chromium	0.80-1.15%
Phosphorus	0.040%	Molybdenum	0.30-0.40%
Sulfur	0.040%	Vanadium	0.20-0.30%

[4] Wakefield, Gene F., "Final Report on Refractory Metal Coatings by Chemical Vapor Deposition," Technical Report AFML-TR-66-397.

The specimen configuration from the above-cited steel is illustrated in Figure 1. Although the overall specimen length is approximately six inches, the reaction zone is only three inches. The specimen tubes were cleaned prior to plating by sand-blasting, wire-brushing, and vapor-degreasing.

Chemicals

Purified grades of the fluorides, WF_6, MoF_6, ReF_6, and VF_5 were obtained from Alpha Inorganics. Tantalum chloride was obtained in the resublimed 99.9% pure condition from Alpha Inorganics. The hydrogen gas was purified by a catalytic hydrogen deoxygenator and a molecular sieve purifier. Commercial grade argon was purified by passing the gas over titanium chips heated to 750°C.

Equipment

The equipment design for vapor-plating is schematically shown in the flow diagram of Figure 2. A photograph of the equipment is shown in Figure 3. Argon from high pressure tanks is heated and passed through a flowmeter. It is then mixed with the refractory metal halide or halides to be reduced. This mixture is then combined with hydrogen in a heated mixing chamber. The mixture of hydrogen, argon, and refractory halide or halides then enters the reaction tube where it passes downward through a heated zone. The refractory metal is deposited on the walls in this heated zone through reduction of the halide or halides by hydrogen. The reaction gases pass through a trap to remove solid by-products and the gases exit through an exhaust system.

Except for two short sections of copper tubing from the gas tanks (hydrogen and argon), the entire system was made up of stainless steel. Thus, an exceptionally gas-tight system was developed to prevent leaks.

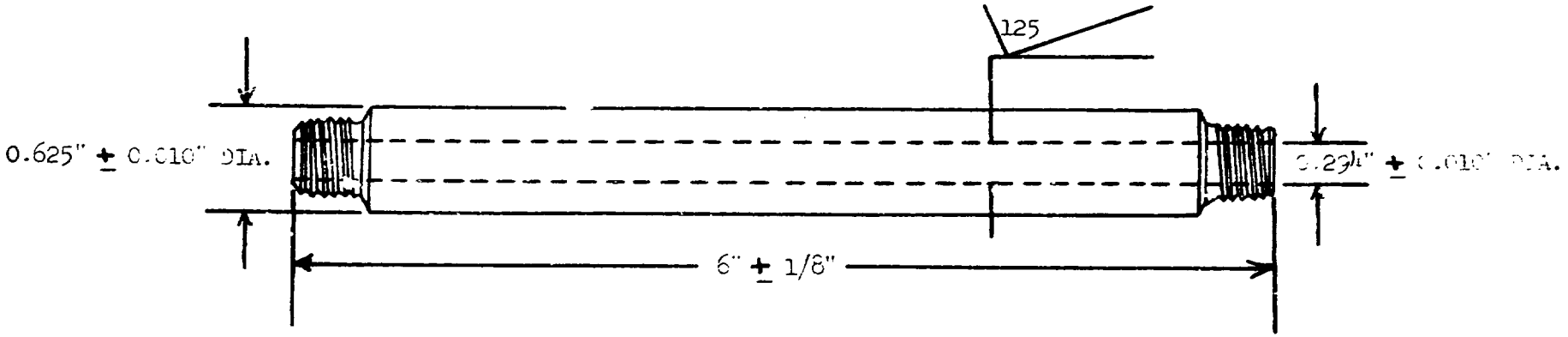
The gas movement was measured with a flowmeter having a claimed accuracy of 3%; the actual flow was calibrated with a wet test meter having a claimed accuracy of 0.5%. The results of the calibration showed a deviation of less than 3% from the smoothed curve values; all of which were within the desired limits.

The mixing chamber, reactant gas cylinders, and the titanium chips cylinder temperatures were controlled by powerstats. Monitoring of the above temperatures was accomplished by the use of a multiple station recorder.

Heating of the specimen tube was done with a Lepel high-frequency induction heating unit. Monitoring of the specimen tube temperature was accomplished by use of an infrared radiation Ircon thermometer. Accuracy of the temperature readings was within ± 10°F.

RESULTS AND DISCUSSION

Feasibility tests involved initial depositions of tungsten onto the



NOTE:

1. 3/4" diameter rough stock, 45° chamfer with 5/8" minimum diameter is permissible.

2. I.D. - O.D. concentric within 0.010".

3. 1/4 NPT, both ends.

FIGURE 1 Chemical Vapor Deposition Test Specimen

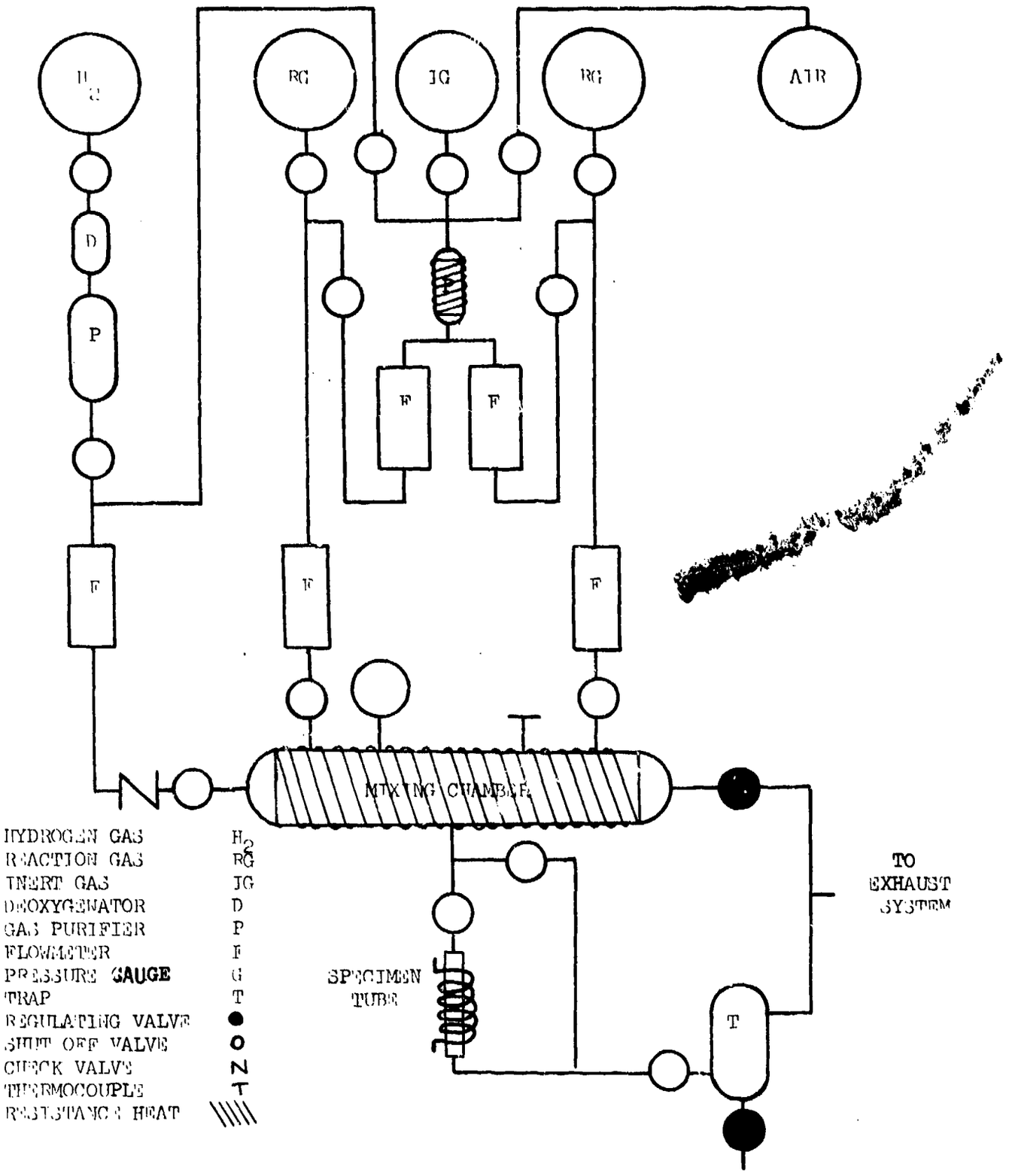


FIGURE 2 Schematic Diagram of
Laboratory Chemical Vapor Deposition System

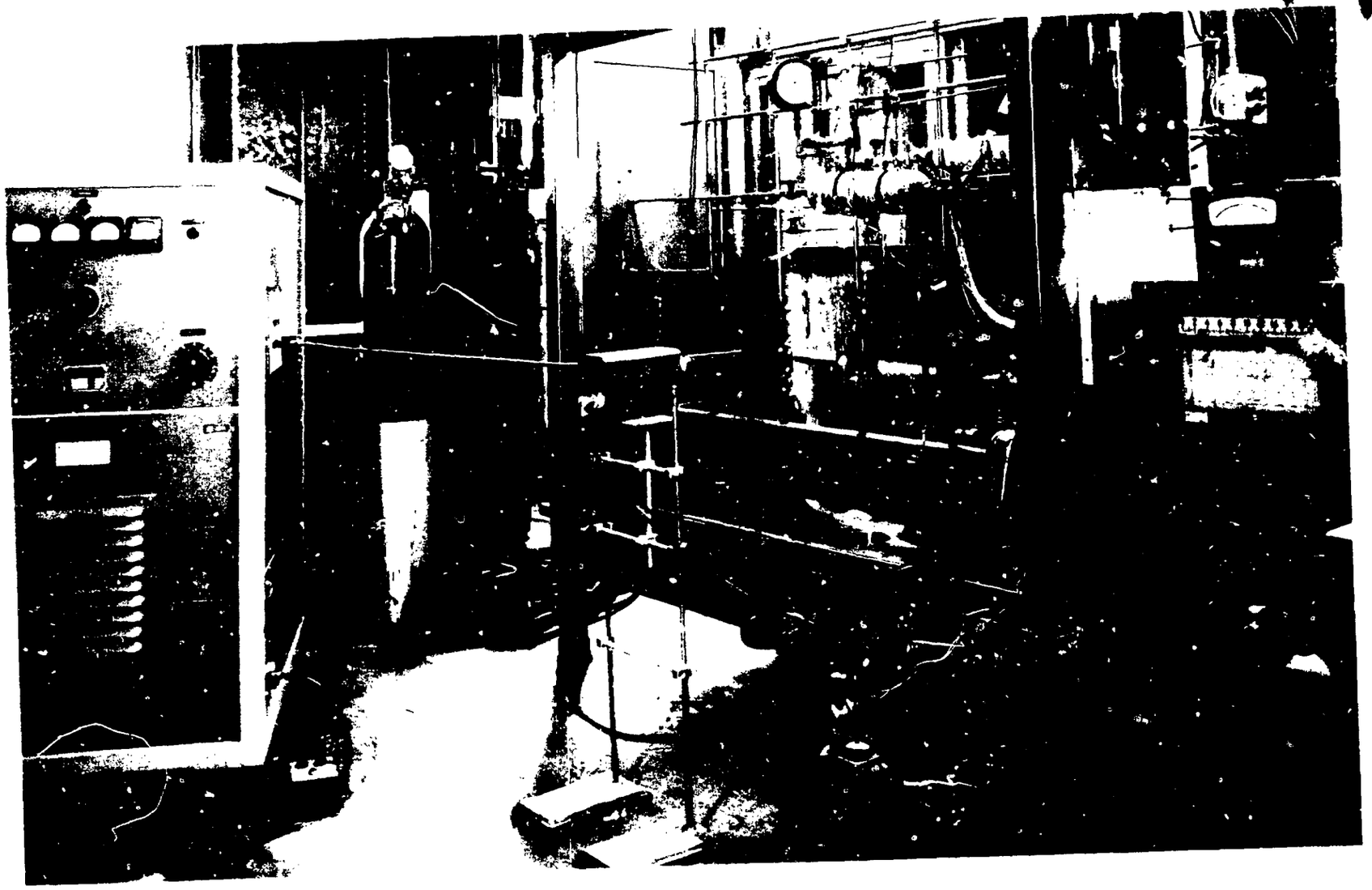


FIGURE 3 Chemical Vapor Deposition Equipment

gun tube bore because of previous success with its deposition.[5,6] Coatings up to 4 mils were deposited on the gun tube specimens at temperatures in the range of 700-850°C. The tungsten coating was a uniform plating consisting of columnar grains growing in a direction normal to the substrate surface. The larger columnar grains start to grow from an equiaxed microcrystalline deposit on the surface. As the thickness of the plating increases, some of the smaller crystals disappear and the diameter of the larger crystals increases. As the grain size increases, the hardness decreases. Photomicrographs of the tungsten coatings on the steel substrate are included as Figures 4 and 5. The steel was etched with nital for 10 seconds to bring out the substrate-coating interface (Figure 4). A Knoop microhardness indentation of the tungsten coating corresponding to a hardness value of 481 is shown in Figure 5.

The quality of the bonding between tungsten and steel substrate was evaluated by a process used originally by Meyer.[1] A cross section at 100X, with microhardness indentations in the interface under loads of 500g, 1000g, and 2000g from left to right is shown in Figure 6. The adhesion of the tungsten coating to the steel substrate was excellent and no separation or flaking was observed.

A photograph of a typical sample obtained when tungsten was plated on the gun tube specimen is shown in Figure 7. A uniform dense coating is deposited in the three-inch reaction zone.

Diffusion of tungsten into the steel substrate was insignificant as evaluated by electron microprobe data. A typical run on the scanning electron microscope equipment to determine the extent of diffusion is shown in Figure 8.

From the data obtained (Table 1), the following optimum conditions were determined for plating tungsten onto the steel substrate:

Temperature	700-850°C
Flow rate	500-1000 cc/min
H_2 to WF_6 ratio	4 to 6
Pressure	1 atm

[5] Powell, C. F., Campbell, I. E., and Gonser, B. W., J. Electrochem. Soc., 93, 258 (1948).

[6] Gehring, J. W., "Tungsten Plating of 25mm Gun Barrels," Technical Report AFATL-TR-72-33.

[1] Meyer, Karl H. and Gehring, John W., "A New Technique for Prevention of Erosion of Gun Barrels," AC Electronics-Defense Research Laboratories, Contract F08635-68-C-0044.

FIGURE 4 Tungsten Coating on Chrome-Moly-Vanadium Steel
Nital Etch 100X

FIGURE 5 Transverse Cross Section with Microhardness Indentation; 500g Load, Hardness R_C 46

Nital Etch 200X

FIGURE 6 Transverse Cross Section with Microhardness Indentations. Loads of 500g, 1000g, and 2000g were applied on Interface to Show Adherence Between Tungsten Coating and Steel

Nital Etch 100X

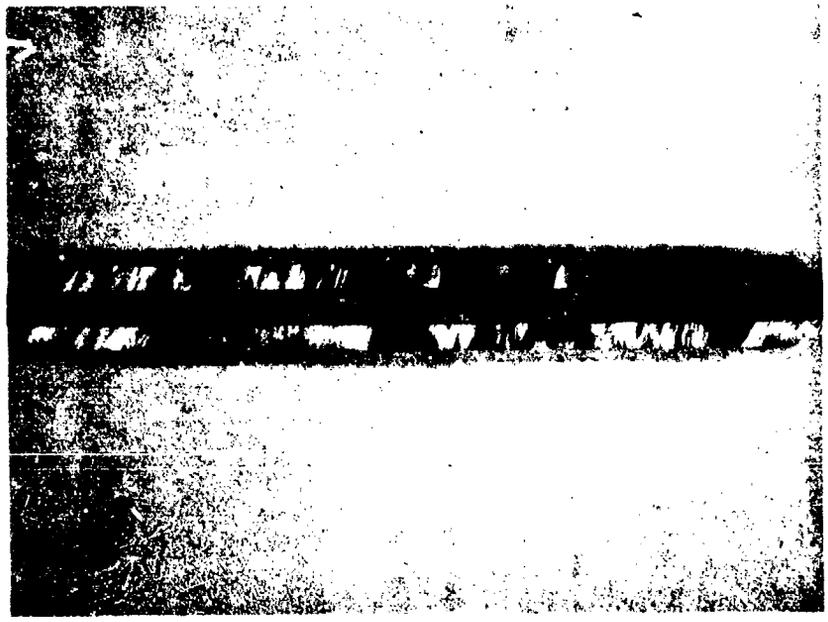


FIGURE 7 Sectioned Tube Showing Tungsten Coating

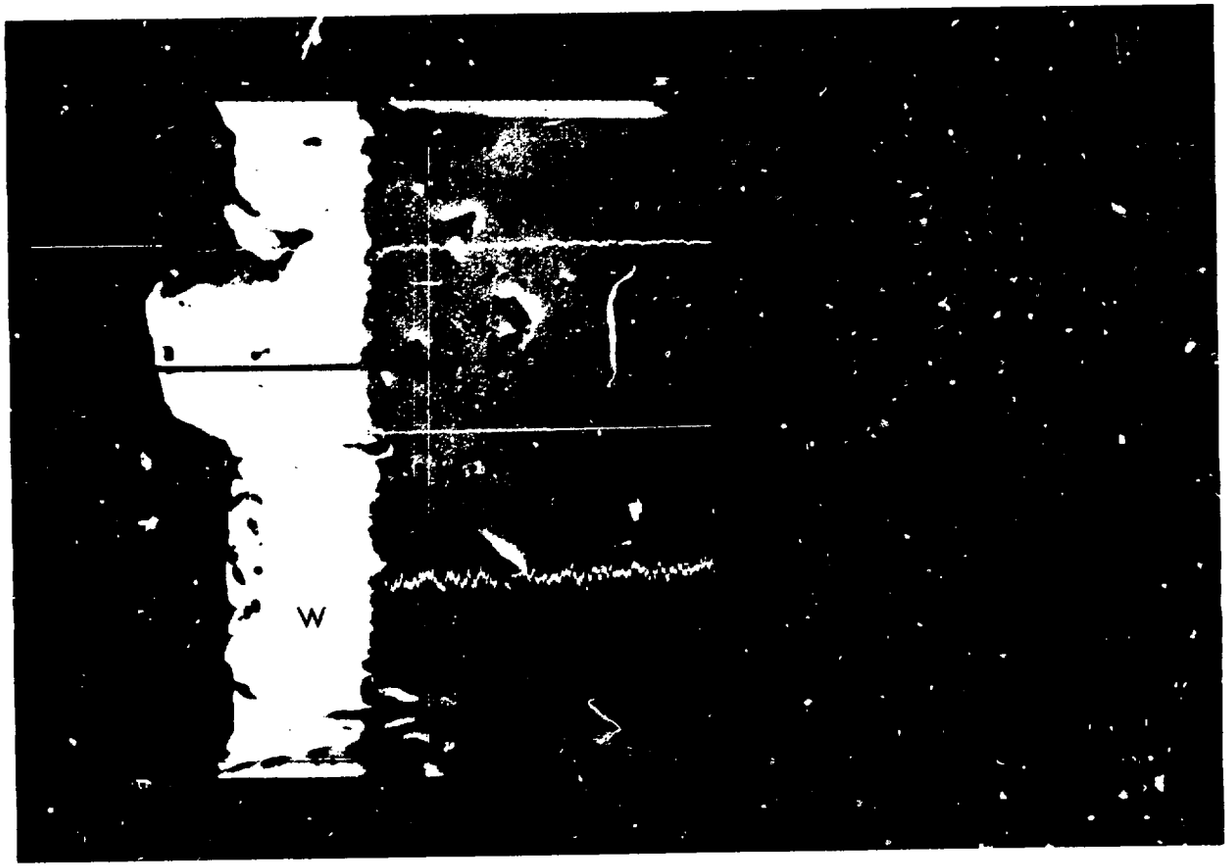


FIGURE 8 SEM Micrograph Showing Extent of Tungsten Diffusion into Steel Substrate

TABLE I. SUMMARY OF TUNGSTEN DEPOSITION

Run No.	H_2 Flow cc/min	Ar Flow cc/min	WF_6 Flow cc/min	Mixing Chamber Temp (°C)	Deposition Temp (°C)	Plate Thickness (mils)	Remarks
1	1200	900	400	230	740	0.4	Thin adherent coating
2	1100	800	150	220	740	0.3	Thin adherent coating
3	1200	400	150	320	755	-	No coating
4	1200	900	200	220	750	0.2	Thin adherent coating
5	1700	900	200	220	750	0.2	Thin adherent coating
6	1200	-	150	230	770	1.3	Good coating, no discontinuities
7,8,9,10,11	1200	-	100	210	750	0.5-0.7	Good dense coating
12	1200	600	100	210	750	-	No coating
13	1200	-	100	220	950	-	No deposit
14	1100	800	170	210	740	1.2	Good adherent coating
15	680	800	100	230	760	2.5	Good coating, 20 min. run
16	960	-	145	220	750	0.6	Thin adherent coating
17	960	800	145	220	730	0.3	Thin adherent coating
18,19	960	1200	145	230	750	-	Poor coating
20,21	960	1750	145	210	740	-	Poor coating
22,23	960	400	145	220	740	-	Poor coating
24	1500	800	150	220	730	-	Poor coating
25	965	-	145	210	760	0.5	Thin adherent coating
26	965	800	145	220	750	-	Poor coating
27	965	100	145	210	750	-	Poor coating
28	1200	270	290	220	750	-	Nonadherent coating
29	965	250	145	220	750	1.0	Good coating
31	1460	250	145	210	750	-	Nonadherent coating
34,35,36,37	965	800	145	220	750	0.5	Thin adherent coating
38,40	965	-	145	220	750	2.0	Good adherent coating
41,42	965	300	145	220	750	0.5	Thin adherent coating
44,45	400	-	60	240	760	0.8	Dense, uniform coating
46	600	-	60	230	750	0.5	Thin, adherent coating
47	240	-	60	240	740	0.6	Good adherent coating
48	400	-	60	240	1020	-	No coating
49	400	-	60	230	850	-	Poor coating
50	400	-	60	240	660	-	Nonadherent coating
54	400	-	60	240	740	1.6	Good coating, 20 min. run
56	965	-	145	210	730	4.0	Good thick coating, 20 min. run
57	965	-	145	210	850	-	Rough coating, 380 KHN
58	965	-	145	220	650	1.0	Nonadherent, 430 KHN
59	965	-	145	210	780	3.0	Good coating, 15 min. run
60	965	-	145	210	830	3.5	Good coating, 20 min. run, 450 KHN
61	965	-	145	220	580	-	No coating
62	965	-	145	220	710	4.0	20 min. run, 470 KHN

NOTE: All the above runs were 10 minute runs unless otherwise denoted in the remarks section.

With the use of the optimum conditions obtained for tungsten plating, the alloys of other refractory metals with tungsten were then attempted. By the alloying of this with the other refractory metals, the ductility of tungsten can be improved.

The first alloy evaluated was a tungsten-molybdenum alloy. Alloys of this system are generally in the W-15Mo range.[7] Coatings of W-20Mo and W-30Mo were obtained by vapor-plating. They were quite thick (~10 mils), but very porous. Problems of vaporizing MoF_6 and clogging within the gas train discouraged any further experimentation of this alloy system.

Rhenium and vanadium depositions were also attempted with tungsten to form alloy coatings. However, the coatings obtained were nonadherent. Higher temperatures than those of the normal tungsten deposition range would be needed to successfully alloy rhenium or vanadium with tungsten. Unfortunately, lack of material forestalled any further experimentation in this system.

Tantalum was, however, plated successfully with tungsten in the form of a Ta-10W alloy. Alloys that were obtained from the tungsten-tantalum plating are shown in Table II. A photomicrograph of a Ta-10W alloy obtained under the optimum conditions for tungsten plating is shown in Figure 9. The coating was a dense uniform adherent plating of approximately 1 mil thickness. Analysis by emission spectrography and microprobe data showed that ten percent tungsten by weight was obtained under controlled conditions. A typically sectioned gun barrel plated with this alloy is shown in Figure 10. Uniformity of the coating was within the limits of ± 5% of the desired thickness. Previous investigators have obtained such alloys only at much higher temperatures (1000°C and above) and reduced pressures. However, this study has shown that such extreme conditions do not have to be used to plate Ta-10W alloy.

From the experimental conditions mentioned earlier for an erosion resistant material, the Ta-10W alloy does satisfy most of the necessary requirements. It exhibits one of the higher strengths for refractory alloys and is also known to have good ductility and toughness compared with other refractory alloys. These properties along with its high melting point, high specific heat, and low thermal expansion make it an excellent choice as a coating for gun barrel bore surfaces.

CONCLUSIONS

Chemical vapor deposition of tungsten on internal bore surfaces of Cr-Mo-V steel tubing was accomplished. Uniform, columnar-grained coatings up to 4 mils in thickness were deposited in the temperature range of 700-850°C. Parametric studies of the deposits were conducted and subsequently applied to alloy deposition. Coatings of tungsten alloyed with molybdenum, rhenium, vanadium, and

[7] Huminik, John, High Temperature Inorganic Coatings, Reinhold Publishing Company, New York, 1963.

TABLE 2. SUMMARY OF ALLOY DEPOSITION

Run No.	H_2 Flow cc/min	Ar Flow cc/min	$TaCl_5$ Cylinder Temp. (°C)	WF_6 Flow cc/min	Deposition Temp (°C)	Thickness (mils)	Remarks
84	950	360	170	30	770	0.7	Good adherent coating Ta-5W
85	1000	360	170	60	770	2.0	2-min. run Ta-45W
86	1000	360	170	40	770	0.8	Ta-10W
87	1000	360	170	30	770	0.8	Ta-5W
88	1000	360	170	40	770	1.0	20-min. run Ta-10W

NOTE: All above runs were 10 minute runs unless otherwise denoted in remark section.

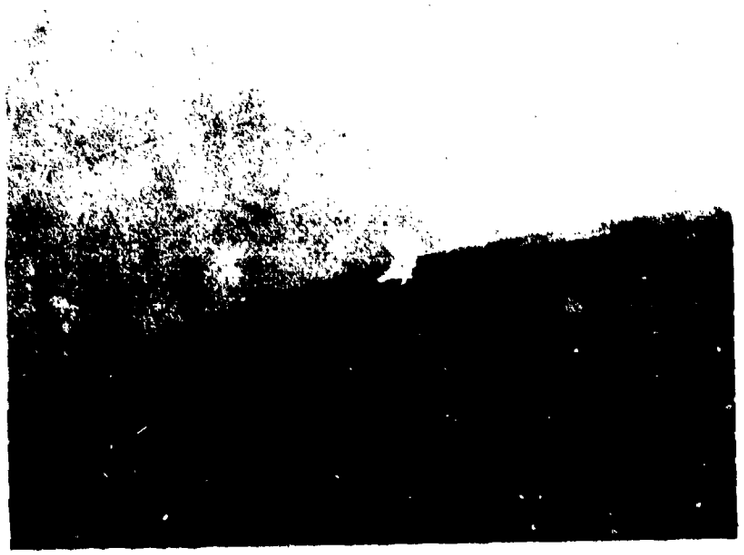


FIGURE 9 Ta-10W Alloy Coating on Chrome-Moly-Vanadium Steel
100X

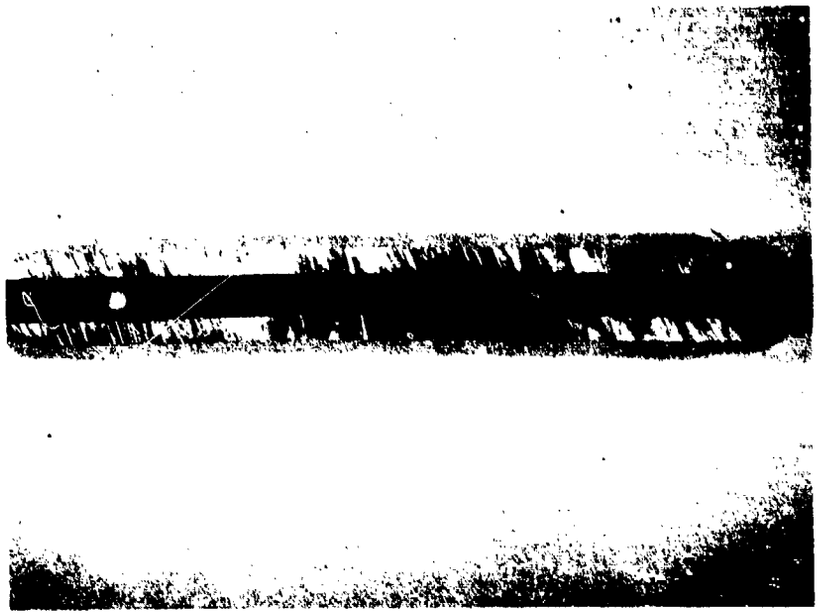


FIGURE 10 Sectioned Tube Showing Ta-10W Alloy Coating

tantalum were plated, respectively. Successful coatings in the case of W-30Mo, W-20Mo, and Ta-10W were accomplished. However, Ta-10W was the only alloy obtained as a dense, uniform, adherent coating.

Plating with Ta-10W coating was feasible at reduced temperatures. The success of plating such an alloy in the temperature range of 750°C is advantageous due to the nature of the steel substrate. By plating at the lower temperature, extensive austenitization of steel is prevented and the rapid quenching of the steel substrate which would be necessary at higher temperature can be eliminated. Consequently, stress changes at the coating interface and property changes in the steel substrate due to phase transformation are minimized.

Future work with CVD should be prefaced by some basic property studies of the tantalum alloy system over a range of 2% W to an excess of 15% W. Determinations of fracture toughness, thermal conductivity, and fatigue strength, as a function of composition, could set a more optimum choice of tungsten content for gun barrel liners. This would allow the designer to specify exact thickness of coatings and substrate steel and to match the proper steel material to the characteristics of the coatings.

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5. Powell, C. F., Campbell, I. E., and Gonser, B. W., J. Electrochem, Soc., 93, 258 (1948).

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THE FORMATION OF REFRACTORY METAL ALLOYS BY SIMULTANEOUS DEPOSITION OF METALS FROM THE GASEOUS PHASE, by A. Crowson

Report R-TR-74-047, Jul 74, 22 p. incl. illus. tables, (DA 1T162105AH84, AMS Code 612105.11.AH84) Unclassified report.

Chemical vapor deposition investigations were undertaken to determine the feasibility of coating refractory metals and alloys onto the inside bores of gun barrels. Coatings of tungsten, molybdenum, vanadium, rhenium, and tantalum as well as tungsten alloys were attempted. Process parameters, compositions, and properties of the coatings were determined. Elemental tungsten depositions were initially used to establish the process parameters for subsequent work

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2. Refractory Metals
3. Coatings
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